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Physical quality and surface hydration properties of wood based pellets blended with waste vegetable oil

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ABSTRACT

To satisfy the demand of reducing extensive usage of wood as a raw material for pellets production, application of alternative raw materials, particularly residues, is necessary. The purpose of this paper is, therefore, to study feasibility of alternative wood pellets with addition of waste vegetable oil and to examine how the oil affects pellet's physical quality, surface properties and pelleting process. Furthermore, a novel approach for estimation hydrophobic properties of biomass pellets was presented. Pellet surface hydrophobicity and water drop absorption rate were assessed by measuring contact angle along time. Oil addition was 2.2% and 5.8% based on dry solids. Pellets were produced by single pellet press method under four compacting pressures (75, 150, 225 and 300 MPa) at three pelleting temperatures (60, 120 and 180 °C). The results show that oil addition significantly increases energy content in wood pellets ($p < 0.05$). Energy content was increased from 19.69 MJ/kg (control) to 20.05 MJ/kg (2.2% of oil) and 20.64 MJ/kg (5.8% of oil). Strength of pellets, as well as sawdust compressibility, was reduced when adding oil. The oil caused lower friction on the pellet–die wall contact area.

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1. Introduction

Waste vegetable oil (WVO) is a residual biomass available in large quantities with high potential for usage as a raw material in solid biofuel production. WVO is non-fossil oil, has high calorific value, low heavy metal content, and low moisture content and does not require any pretreatment [1]. The main focus of this paper is valorisation of waste vegetable oil (WVO) by using it as a raw material in production of wood based pellets. The aim of paper is to examine how the WVO addition will influence physical pellet quality, pellet surface properties and pelleting process. In addition, a novel approach to assess pellet hydrophobicity by contact angle measurements is presented. The paper answers in detail how the oil addition changes compressibility of sawdust, strength of pellets, where are the limits of densification, how the pellet hydrophobicity changes by adding WVO and how hydrophobicity can be more quickly evaluated by measuring contact angle on compacted powder. The main challenges related to pelleting of oily sawdust are discussed.

The EU is currently a leader in world pellet consumption (70% of world consumption) [2]. Demands for pellets in Europe have already exceeded their production; thus significant amounts of pellets have been imported mainly from US and Canada, although the production capacities in EU are not fully used [2–5]. Moreover, European Commission predicts that pelleted solid biofuels will play an important role in meeting renewable energy targets in Europe [4]. All of these indicate a further growth in pellet production. Forest biomass is almost exclusively used as a feedstock for pellet production. In order to provide sustainable production, the goal is to use new and cheap raw materials available in large quantities, which will partially replace wood. The usage of by-products or waste from industry, agriculture or households is the best way to achieve environmentally and economically sustainable fuel production. Utilization of bio-residues is preferable not only from the aspect of using these materials for energy generation, but also from the aspect of avoiding the problems related to waste disposal.

WVO is discarded by restaurants, food manufacturers (potato chips, breaded fish sticks, doughnuts, etc.) and other facilities (schools, hospitals, households, etc.) because it cannot be further used in human or animal nutrition. Estimation is that total EU potential is 3.55 million tonnes per year which is about 8 l per capita/year [6]. Inappropriate disposal of WVO can be environmentally hazardous; therefore its further utilization is preferable. Until 2002 most of the collected WVO was used as a feed component, but EU banned its application because of harmful components formed during frying which can return into the food chain trough the animal meat [7]. Thus the application of WVO for energy generation would be a good form of its utilization.

WVO addition can improve wood fuel properties, but densification of wood with added oil can be more challenging due to inhibition of the main binding mechanisms for densification and lowering friction in the die [8]. Vegetable oils are reported in literature as an additive

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which reduces die wall friction and energy consumption for pelleting process due to lubricating effect [9]. To our knowledge, blending waste vegetable oil with wooden biomass has not been previously reported in pellet production. A recent work of Shang et al. [10] showed that rapeseed oil addition reduces static friction on the die wall and strength of pellets produced from torrefied wood chips. Ståhl and Berghel [11] studied how the addition of different amounts of rapeseed cake which contained 18% of oil affects energy consumption and some mechanical properties of wood based fuel pellets. The authors concluded that energy consumption, durability and bulk density of pellets were reduced with increased amount of rapeseed cake. Some commercial pellet producer has already started with production of pellets covered with thin oil layer aiming to reduce dust content [12].

Surface characteristics and water absorption rate of pelleted solid biofuels are important quality parameters especially when it comes to outdoor and indoor humid storage conditions. A common method for evaluating biomass fuel hydrophobicity is based on measuring the material moisture absorption capacity, when surrounding temperature and humidity are constant [13–15]. The method is reliable, but time consuming, because it requires equilibration of moisture in the material and surrounding atmosphere, which can take several days. The method presented in this paper is based on measuring contact angle (θ) and absorption rate of a water drop placed on a compacted wood powder. The method is fast and particularly convenient for densified biomass like pellets or briquettes. Also this method can give an information about material/pellets behavior in direct contact with water which can replicate outdoor (rain) and indoor (condensation) storage situations. Similar measurements have been performed in compacted powders of soy and wheat flour [16,17] and pharmaceuticals [18,19], but this approach, to our knowledge, has never been used for testing the surface properties of densified solid biofuels.

In order to study the effect of WVO on pelletability and pellets physical properties the oil has been added in two different amounts (2.2 and 5.8% on dry solids). Blends and control pellets (no oil addition) were produced by single pellet press method at three different temperature (60, 120 and 180 °C) under four compacting pressures (75, 150, 275 and 300 MPa). Pellets for the θ measurements were produced at 120 °C under a pressure of 300 MPa.

2. Material and methods

2.1. Preparation of the raw materials

Spruce stumps (*Picea abies*) were selected as raw material and cut from a local forest in Ås, Norway in December 2012. In order to preserve woods physicochemical properties the stumps were stored in a freezer until further usage. Prior to the pelleting the wood was defrosted, the bark was removed, stumps were cut in smaller pieces, pre-dried and grinded in a hammer mill (Brabender, Duisburg, Germany) with screen opening size of 1.5 mm. Wood pieces were pre-dried at 105 °C in order to make material more brittle and thus easier to grind by hammer impact. Adopting literature recommendation for pelleting process [20], the wood powder was afterwards moisturized to 10 ± 1% moisture content by spraying water in an intensive laboratory mixer (Diosna P1/6, Germany). The final moisture content in spruce powder was 10.8 ± 0.3% (Table 2). This material served as control sample and as a base raw material for the blends with WVO. The moisturized powder was stored in vacuum packed plastic bags at ambient conditions until further usage (1–2 weeks).

The WVO was obtained from McDonald's restaurant in Vestby, Norway. Before spaying, the WVO was filtrated trough filter paper. Basic physicochemical properties of the WVO are presented in Table 1.

Two levels (L_1 and L_2) of WVO were added to the powder by spraying in a high shear mixer having three impellers and a tulip-form chopper (Diosna P1/6, Germany). An intensive mixing (mixer 250 rpm; chopper 500 rpm), manipulating oil drop size in the spraying lance (Düsen-Schlick GmbH, Germany, Model 970) and a preheating of the WVO to 60 °C were used to facilitate an even distribution of the oil in sawdust. Final content of oil in sawdust was calculated from the difference in the higher heating value (HHV) of control powder and powder with oil. Blended powders L_1 and L_2 contained 2.2% and 5.8% of WVO based on dry solids, respectively. The materials were pelletized at least 48 h after the mixing in order to allow an eventual penetration of WVO into pores.

Table 1
Properties of WVO.

Property	Value
HHV (MJ/kg)	37.60 ± 0.07
Density[a] (kg/m^3)	910.7
Dynamic viscosity [21] (Pa s)	0.117 (at 20 °C) 0.026 (at 60 °C) 0.007 (at 120 °C) 0.004 (at 178 °C)
Ash content [22] (%)	0.006

[a] Measured at room temperature.

2.2. Characterization of the raw materials

Following analyses on raw materials were performed:

- The particle size distribution of spruce sawdust was measured by Mastersizer 3000 optical unit combined with Aero S dry dispersion unit (Malvern Instruments, U.K.).
- The bulk density was determined by measuring the mass of a known volume of material that has been loosely poured into a graduated cylinder. The density of oil was determined by measuring the mass of know volume of oil in a graduate cylinder (±0.2 ml) on analytical balance (±0.0001 g)
- The water activity value (a_w) was measured by a Rotronic HygroLab C1 (Switzerland) instrument. Average temperature during the a_w measurements was 20.16 ± 0.1 °C. a_w value describes availability of free water in different products (typically food and feed). Free water affects products microbiological, enzymatic and chemical stability.
- The moisture content was determined by drying in an oven for about 20 hours (Termaks, Norway) at 105 °C (EN 14774-1).
- The content of ash and volatile matters was determined in a muffle furnace (Nabertherm D-2804, Germany) according to EN 14775 and EN 15148 standard procedure, respectively.
- The fixed carbon ($\%_{d.b}$) was calculated by difference between 100 and the sum of the volatile matter and ash content.
- The HHV was calorimetrically determined (Parr 1341 Oxygen Bomb Calorimeter, Moline IL) following the EN 14918 standard procedure.

2.3. Pellet production

Pellets were produced by single pellets press method in a pelleting unit that consists of a steel cylinder with a concentrically positioned compressing channel of 8 mm in diameter. A jacket heater (450 W) is temperature controlled by a PID (proportional-integral-derivate). A tungsten carbide pressing rod was used for compression. The compressing force was applied to the rod using an Instron 5800R 100 kN universal compression-tension testing machine (England). Pelleting unit was designed and built at the Norwegian University of Life Sciences (Ås, Norway). Pellets were produced following the same procedure as described in Mišljenović et al. [14], based on previous work of Salas-Bringas [23]. Briefly summarized, when set temperature was reached, the die channel was filled with material and the pressing rod was placed into the die. After 3 min of tempering, the biomass was compressed at a rate of 2 mm/min until the set pressure was reached. Afterwards the pressure was released, the bottom rod

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removed and the pellet was pressed out from the channel. The pellets were produced at 60, 120 and 180 °C under four compacting pressures—75, 150, 225 and 300 MPa. A wide range of pelleting temperatures and pressures was selected to examine the physical behavior of the ground materials and pellet quality under different conditions that can occur during regular pelleting. The pressure required to initiate pellet discharge from the die i.e. the pressure at incipient flow (p_{max}) was recorded to determine differences generated by the friction on 'die–pellet' contact area. The discharging speed of the rod was set to 15 mm/min and attention was paid to avoid p_{max} exceeding the compacting pressure. The produced pellets were stored in sealed plastic bags at ambient conditions until further testing.

2.4. Testing the pellets

The strength of pellets was measured 48 h after production. Before the strength measurement, dimensions (length and diameter) and mass of each pellet were measured to calculate pellet density. The strength of the pellets was measured by a diametric compression test following the same procedure as described in Mišljenović et al. [14]. The strength measurement was performed by a diametric compression test using a 60 mm diameter probe connected to Lloyd LR 5 K texture analyzer (Lloyd Instruments, U.K.). The maximum normal force at pellet breakage was recorded. Compression tests were performed with speed of a 1 mm/min.

2.5. Contact angle (θ) and scanning electron microscopy (SEM) measurements

In order to compare surface hydration properties of porous compacted materials, θ measurements were performed on pellets. For the θ measurements the materials were compressed in 8 mm diameter pellets at 120 °C under a pressure of 300 MPa. The θ measurements were conducted at room temperature with a video based optical θ measuring device OCA 15EC (DataPhysics Instruments GmbH, Germany) (Fig. 1). Defined volume of distilled water (5 µl) was disposed from a dosing syringe on the upper plane surface of pellet and video of drop absorption was recorded. θ and its changes with time were calculated by SCA 20 software. Apparent rate of water absorption was calculated as a slope of linear relationship between θ and time at linear period of water absorption.

To examine the effect of pellet porosity on water absorption rate, the pellet surface was observed by SEM. Pellets were attached to aluminum stubs with carbon based glue and the upper surfaces were coated with gold palladium in a Sputter coater (Polaron SC 7640). Imaging was performed by using Zeiss EVO 50 EP SEM operated at 20 kV acceleration voltage and 30 pA of beam current. Two pellets of each material were prepared at 120 °C under a pressure of 300 MPa. The whole surfaces were scanned at different magnifications, and representative images were selected and presented in the paper.

Table 2
Raw material properties.

Parameter	Control	L_1	L_2
Bulk density (kg/m^3)	119.44 ± 1.54^a	119.35 ± 1.95^a	122.05 ± 1.81^a
a_w value	0.517 ± 0.009^a	0.540 ± 0.007^a	0.461 ± 0.018^b
Proximate analysis			
Moisture content (%)	10.8 ± 0.3	10.6^*	10.2^*
Ash (%$_{d.b.}$)	0.28 ± 0.02^a	0.27 ± 0.05^a	0.28 ± 0.04^a
Fixed carbon* (%$_{d.b.}$)	12.14	8.19	7.62
Volatile matter (%$_{d.b.}$)	87.58 ± 0.75	91.54 ± 0.15	92.10 ± 1.08
HHV (MJ/kg $_{d.b.}$)	19.59 ± 0.16^a	19.99 ± 0.10^b	20.64 ± 0.18^c

^{abc}Different letters within the same row indicate significant differences according to post-hoc Tukey's test (95% confidence interval). *Calculated values.

2.6. Data analysis

The experiment was designed around three factors: oil content (control—0%; L_1—2.2%; L_2—5.8%), compressing pressure (0, 75, 150, 225 and 300 MPa) and temperature (60, 120 and 180 °C). Minimum 3 replicates were tested per each combination of the factors. One-way analysis of variance was used to test if the HHV, a_w value, bulk density, and ash content (responses) differed between different types of materials (factor). The effects of oil content, pressure and temperature on pellet strength, pellet density and P_{max} were examined using General Linear Model ANOVA ($p < 0.05$). Tukey's HSD tests were employed to determine which groups differed (95% confidence interval).

3. Results and discussion

3.1. Characterization of the raw materials

The addition of WVO caused changes in the ground spruce sawdust. The powders with WVO were less dusty, slightly darker and flowed less during handling and filling of the die channel. The changes in powder flow behavior are particularly important for potential industrial application of the oily raw materials. Good flow of powders can be achieved by careful design of silo and equipment or by modification of powder flow properties [24].

The most important properties of tested materials are presented in Table 2. The bulk density varied between 119.35 and 122.05 kg/m^3. The oil addition did not cause significant changes in the bulk density of spruce sawdust ($p < 0.05$). The a_w value of tested materials was significantly reduced ($p < 0.05$) only when the WVO was added in higher amount (L_2). This can be explained by covering the water with oil layer and making it in that way not accessible for the measurement.

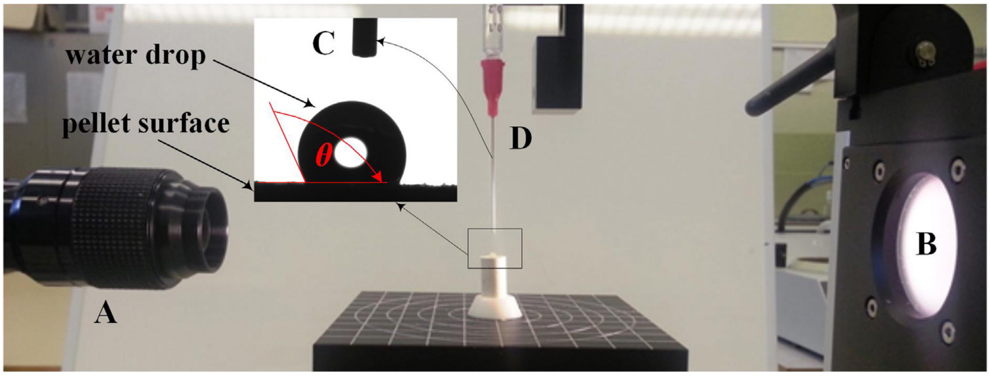

Fig. 1. Experimental setup for the θ measurement. Letters indicate items as follows: A—camera; B—light source; C—image of a drop on top of a pellet surface for θ tests; D—dosing syringe with a needle.

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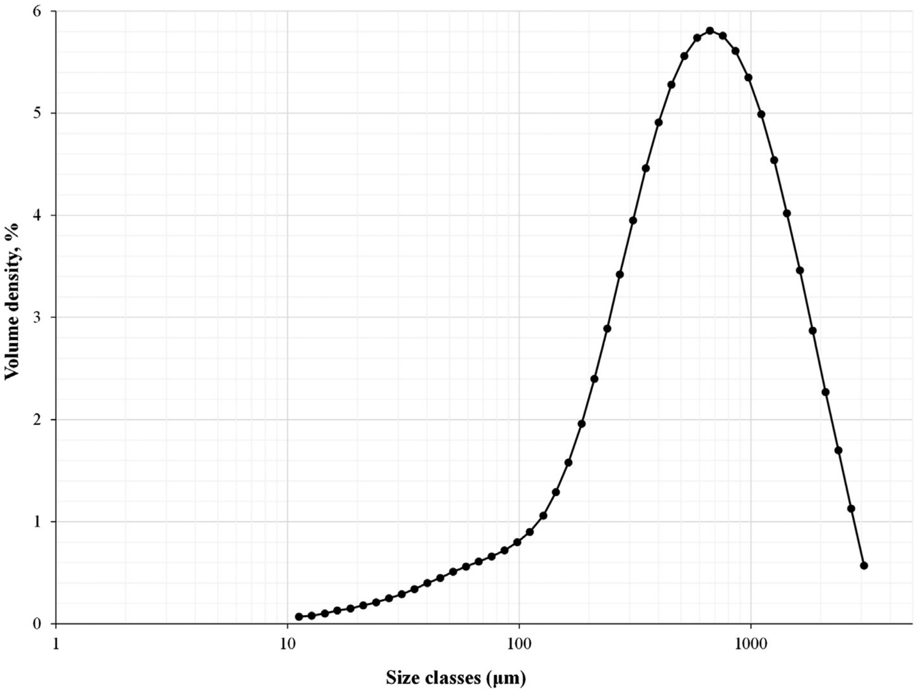

Fig. 2. Particle size distribution of spruce sawdust.

The a_w values of tested powders were very low indicating stability of materials with respect of their microbial activity. Microbiological activity can be a reason of material self-ignition, deterioration of material and health problems. In this range of a_w values (0.461–0.540) growth of microorganisms is not possible [25].

The proximate analysis of powder showed expected changes such as increasing of the volatiles content with WVO addition and no significant change of the ash in powder. Tested powders had low ash content. The most important change caused by WVO addition is reflected in increased energy content. Significant increasing was observed ($p < 0.05$). Energy content was increased from 19.69 MJ/kg (control) to 20.05 MJ/kg (L_1) and 20.64 MJ/kg (L_2). Compared with other method of biomass energy fortification like torrefaction, the addition of 2.2% oil increased the final energy content in the powder similarly as torrefaction at 225 °C [14]. This way of increasing the energy content is definitely easier and less energy demanding compared to torrefaction.

In addition to the tabular data which describes the raw materials, the volume based particle size distribution of spruce powder is presented in Fig. 2. 90% of the particles had diameters lower than 1750 µm and 10% had higher than 166 µm. Ground particles in commercial pellet production are typically coarser than the ones used here. Finer grinding was chosen because the single pellet press does not simulate the effect of the 'second grinding step' that normally occurs when material shears

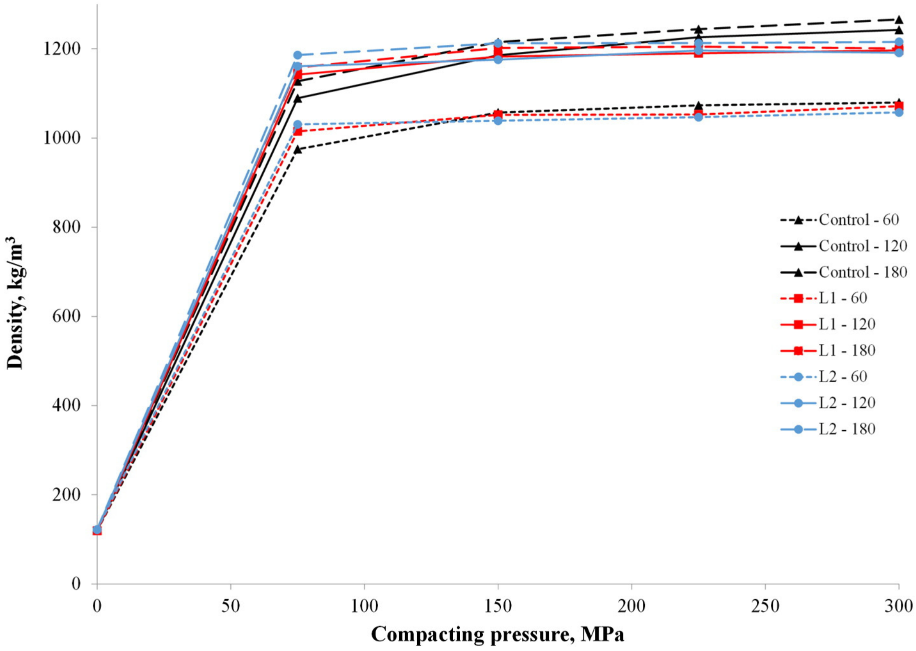

Fig. 3. Compressibility of spruce sawdust with different oil levels at different die temperatures.

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between die and rollers. The mean volume diameter of particles D [4;3] was 819 μm.

3.2. Pellet physical properties

Pellets produced in the single pellet press have a well defined cylindrical shape. In this experiment the control pellets were well-formed, with shiny and smooth surface, while the L_1 and L_2 pellets had more porous structure and could easily disintegrate, indicating poor binding between the particles. In this paper physical characterization of the pellets was performed by measuring their density and strength.

Fig. 3 presents a compressibility curve for the three tested materials. Density of the pellets increased with increasing pelleting temperature. Pellets made at 60 °C were significantly less dense then pellets made at higher temperatures (120 °C and 180 °C) because lignin does not soften at 60 °C; thus its binding ability possibly was not well activated. When softened, lignin diffuses between the particles and after cooling of pellets, it hardened forming solid bridges that bind the particles together [26]. Differences between the pellets made at 120 and 180 °C were not pronounced.

Increasing the compacting pressure increase pellet density, especially at the first stage of compression when particles rearrange and pack closely, pressing the air trapped among particles to flow out. During this stage particles do not change shape and size. With further pressure increase, compaction rate decreases, particles deform and therefore contact surface for their binding increases. In the last stage of compression small changes in the density can be noticed because the material is approaching its true density (no air pockets). After the compression typically relaxation of pellet occurs. This is known as “spring back effect” which happened because of expansion of remained compressed air and/or the relaxation of elastic deformation [27].

The oil addition changed sawdust compressibility. The densities of control pellets significantly increase with increasing compacting pressure from 75 to 300 MPa, which was not the case for L_1 and L_2 pellets. Densities of the oily materials (L_1 and L_2) did not differ, but both differed from control material. The results show that WVO made the material less compressible, probably because of reduced interparticle friction and poorer binding among the particles. Additionally, L_1 and L_2 lines overlap indicating that the amount of oil between 2.2 and 5.8% did not have an influence on pellet density. Control pellets had higher density than L_1 and L_2 pellets except for those produced at 75 MPa.

The results of diametric compression tests are presented in Fig. 4. The peak force at breakage was recorded and the results are expressed as the maximum force per length of pellet (N/mm). Temperature of the die during compaction affects the pellet strength. Stronger pellets were produced when temperature was higher. The effect was particularly clear in case of control pellets (Fig. 4). The strongest pellets were produced from pure spruce at 180 °C (39.1 ± 1.6 N/mm). Effect of higher temperature was not so pronounced in case of oily pellets. The strength of pellets decreased with addition of WVO indicating poorer binding among particles. Binding mechanisms during pelleting can be divided in five groups: solid bridges, adhesion and cohesion forces, surface tension and capillary pressure, attraction forces between solids and interlocking bonds [27]. The oil layer on the sawdust particles led to the surface deactivation, which resulted in deactivation of all the mechanisms that include contact sites and attraction forces between surfaces. Similar effect was observed by Nielsen et al. [28] in sawdust with higher extractive content which deposition on particle surface deactivated their binding ability. Since the sawdust particles were covered with oil, effect of lignin softening and diffusion among particles did not contribute in a great extent to particle binding. Only interlocking and capillary attraction between the particles due to entrapped oil gave the strength to pellets. Particle interlocking was probably poor because of the oil's lubricating effect which can increase sliding between the particles. Capillary attraction due to oil layer is lower compared to water since the force is directly proportional to surface tension [29]. Surface tension of oil is about half of water value and decreases with increasing temperature [30].

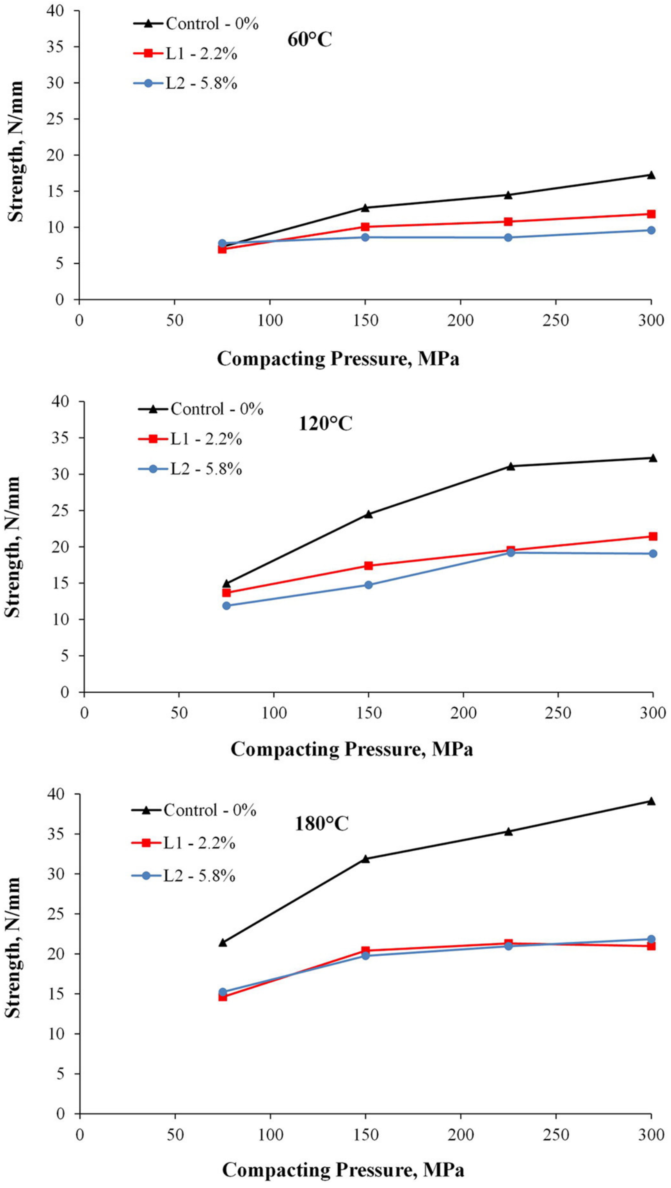

Fig. 4. Strength of pellets produced at different compacting pressures and temperatures.

3.3. Pellet surface properties

Water absorption was evaluated by changes in θ over time. The compaction of powders is a critical step for this measurement because it can alter the surface of pellet and porosity of pellet and in a way not represent the original powder properties.

Clear difference in initial θ and water absorption rate between control and oily samples was observed (Table 3). According to the values of initial θ all surfaces can be considered as hydrophobic ($\theta > 90°$).

Table 3
Initial θ and apparent rate of water drop absorption rate.

Material	Initial θ (°)	Rate of water drop absorption (°/s)
Control	117.0 ± 6.6^a	2.1 ± 0.5^a
L_1	100.2 ± 2.0^b	14.7 ± 1.9^b
L_2	98.5 ± 2.4^b	14.7 ± 2.7^b

^{a,b}Means that do not share a letter are significantly different ($p < 0.05$).

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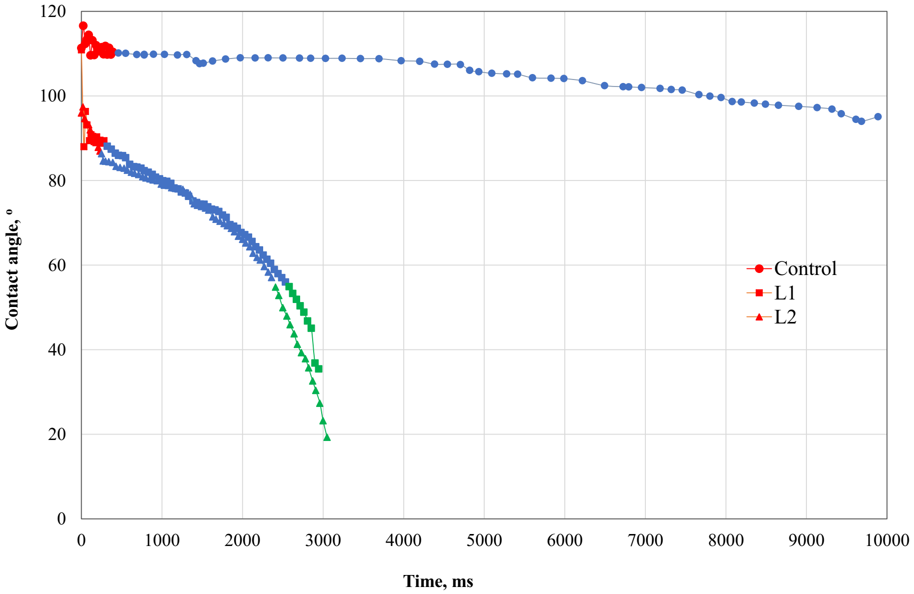

Fig. 5. Changes in θ of water drop deposed on pellet surface as a function of time (red colored marker—zone A; blue colored marker—zone B; green colored marker—zone C). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Although it was expected that the WVO will make material less hygroscopic, the results showed opposite. The pure wood absorbed water drop slower and initial θ of drop was higher (Fig. 5 and Table 3). The difference can be easily observed also from Fig. 6 where the drops on the top pellet surfaces at different materials and different time intervals were presented. After 2.4 s water drop is more absorbed for the L_1 and L_2 samples, while the control one is still well-formed on the pellet surface. This can be attributed to differences in binding between the particles. As already mentioned, the pure wood particles bind better so porosity of the pure wood pellets was lower, making more difficult for water to penetrate among particles. The water absorption trough the pellet surface involves: 1) intra-particle diffusion, meaning the penetration of water inside the wood fibers; and 2) inter-particle diffusion, meaning the penetration of water trough voids between wood particles. The first path can be limited due to collapsing of fibers exposed to high pressure during compaction. The second path depends on level of compaction and particle binding capability which can be correlated with the porosity of compacted surface. Images of surface analysis by using SEM were presented on Fig. 7. The surface of control pellets is more homogenous, with fewer pores, indicating better adhesion between the particles. The L_1 and L_2 pellets have visible inter-particle gaps and voids, with visible fiber layers which explain easier water penetration in the pellets.

When analyzing videos, a similar pattern in water absorption rate was observed. Three zones in water absorption process can be distinguished. Zones are pointed as A, B, and C on Fig. 5 for the L_1 and L_2 absorption lines. Zone 1 can be considered as 'drop settling' zone, which is characterized by large oscillation in θ value. In this zone, right after placing the drop on the surface, the drop is still moving due to inertia. Zone B is 'linear absorption' zone, characterized by linear decreasing in θ value. Apparent rate of water absorption was calculated from the linear part of absorption curve (B). Zone C or 'swelling zone' is

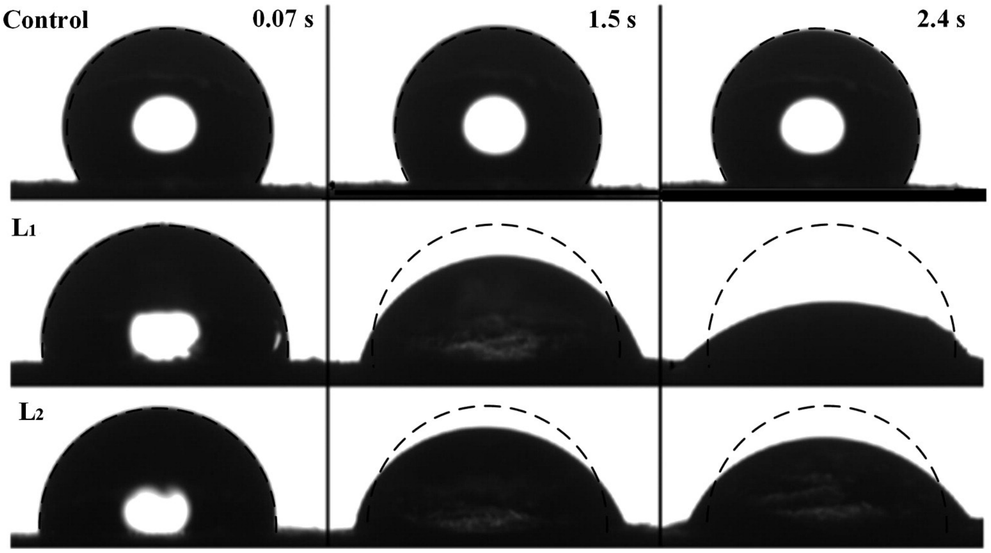

Fig. 6. Sessile drop on the top pellet surface at different time intervals. Dash line represents initial drop profile.

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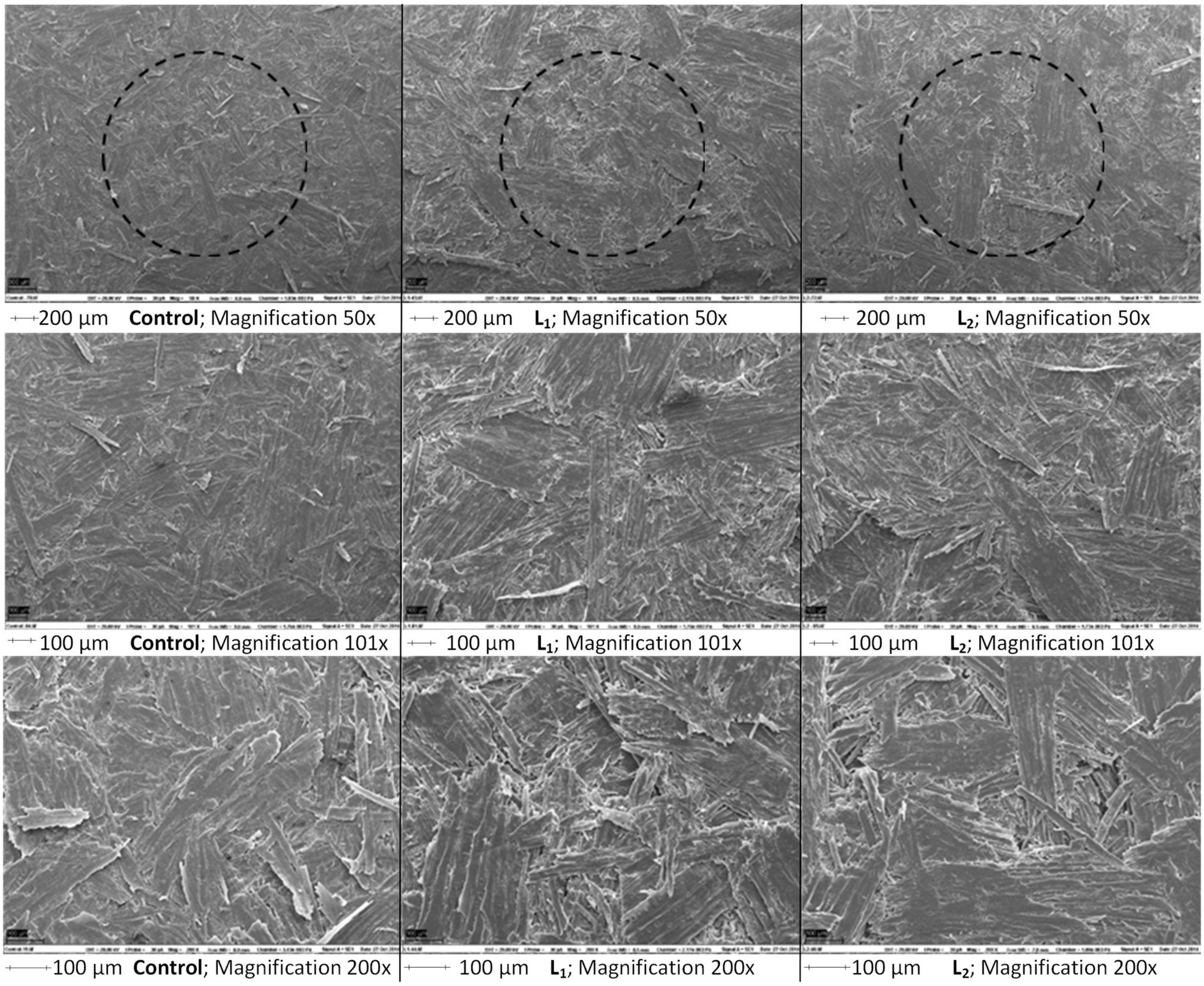

200 µm **Control**; Magnification 50x
200 µm **L_1**; Magnification 50x
200 µm **L_2**; Magnification 50x
100 µm **Control**; Magnification 101x
100 µm **L_1**; Magnification 101x
100 µm **L_2**; Magnification 101x
100 µm **Control**; Magnification 200x
100 µm **L_1**; Magnification 200x
100 µm **L_2**; Magnification 200x

Fig. 7. SEM of top pellet surface at lower (a–c), middle (d–f) and higher magnification (g–i). The control, L_1 and L_2 pellet are presented in the same raw, respectively. The dash circle represents an average drop base area.

characterized by intensive swelling and disintegration of pellet surface. In this zone the contact between drop and solid surface is unclear.

3.4. Effect of WVO addition on friction in the die

Pelleting in single pellet press is a method that simulates compaction that occurs inside of a die hole in flat or ring die pellet press. This method has been used for testing material pelleting i.e. compacting properties and can be considered as first step for process and experimental design. It cannot be a replacement for pilot or large scale experiments because it only replicates the process of compaction inside the die hole. Energy consumption cannot be measured in single pellet press but can be indirectly estimated [14,31]. In this paper the pressure required to initiate pellet discharge (p_{max}) was used as an indicator of the friction generated in the die–pellet contact area. During pellet discharge, p_{max} never exceeded the value of the pressure used to produce pellets. Absolute value of this parameter does not indicate the extent of the power requirements in larger processes, but it can show the difference between materials and rank how easy or difficult material will flow through die. The material's resistance to be moved in a die hole is in large extent responsible for the power requirements and throughputs in commercial pellet presses.

Results showed that p_{max} values were reduced by oil addition and temperature increase. Moreover, higher oil content led to a lower p_{max}. This is a consequence of the lubricating effect of WVO. These findings are in agreement with those presented in work of Ståhl and Berghel [11] and Shang et al. [10] who both performed pelleting in pilot scale pellet press. p_{max} was higher when higher pressure was used for pelleting (Fig. 8). Lower friction in the die–pellet contact area will reduce the material retention in the pellet press, which will increase the material throughput or overall capacity of pelleting line.

During pelleting, it was possible to observe some oil retention on the bottom rod and on the bottom part of the single pellet press. Also, pellets produced at higher pressure had an 'oil gradient' which can be a result of pressing out the WVO from fiber lumen and inter-particle space (Fig. 9). The leakage can be a reason why the difference between L_1 and L_2 pellets at 180 °C was smaller compared to pelleting at other two temperatures. The viscosity of the WVO at 180 °C is very low which facilitate the leakage (Table 1). In context of real pellet press where material continuously flows through die 'oil gradient' probably would not appear. For a large scale production the main concern is about the previously mentioned powder flowability and the fact that temperature in the die is generated by friction between material and the wall, so if the friction is too low temperature will not be high enough to ensure pellet formation and good pellet quality. This article shows the effects of oil in wooden materials, but an investigation in a pilot or commercial pellet press is needed to find the optimum between processing and physical quality.

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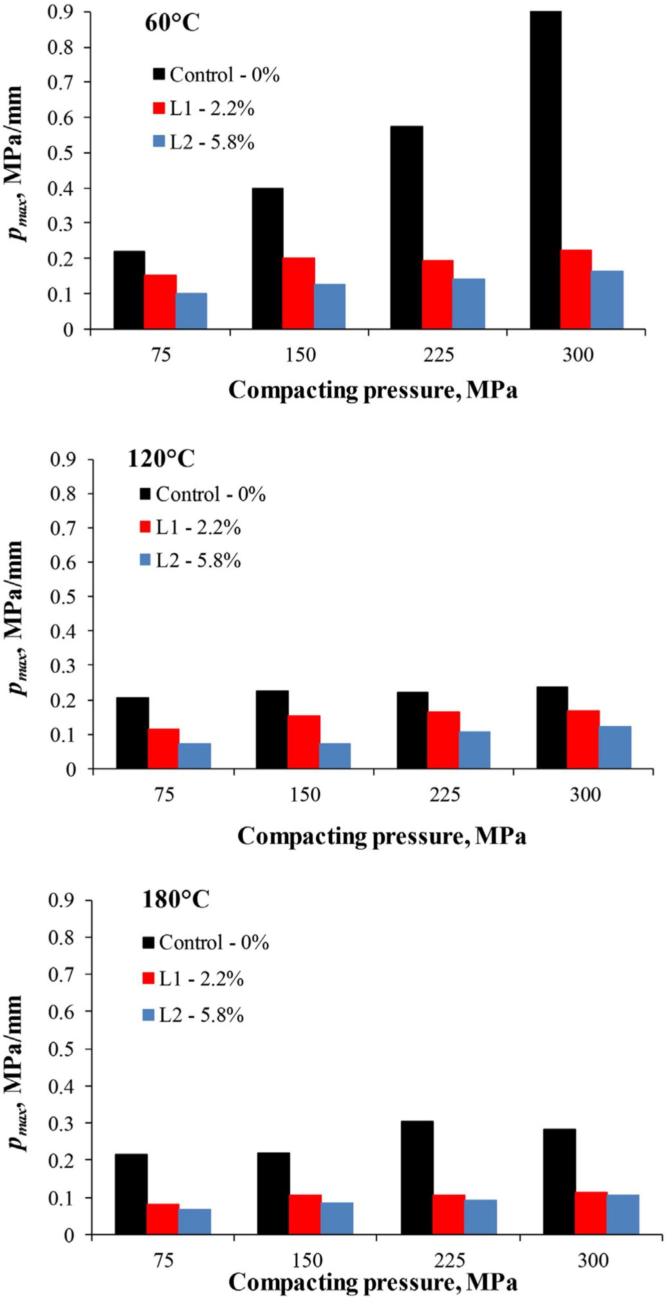

Fig. 8. Discharging pressure (p_{max}) for pellets produced at different compacting pressures and temperatures.

4. Conclusions and recommendations

Oil addition increased energy content in biofuel pellets and reduced friction on the pellet–die contact area indicating possible reduction of energy demands for pelletizing. However, compressibility of the sawdust and pellets strength was reduced with WVO addition. A compressible behavior of the blends was found when compacted at low pressure, and a non-compressible behavior was found at high compacting pressures. By measuring contact angle and its changes with time, it was possible to estimate pellet hydrophobicity and how the materials will behave in direct contact with water, situation that can happen during wet storage conditions (rain and condensation). The method proved to be faster compared to conventionally used methods to test hydrophobicity. Higher porosity of oily pellets caused a faster absorption of a sessile water drop compared to pure wood. From the practical point of view, future studies should incorporate pilot scale testing and finding optimal process parameters in order to compromise pellet quality and energy requirements for pelletizing.

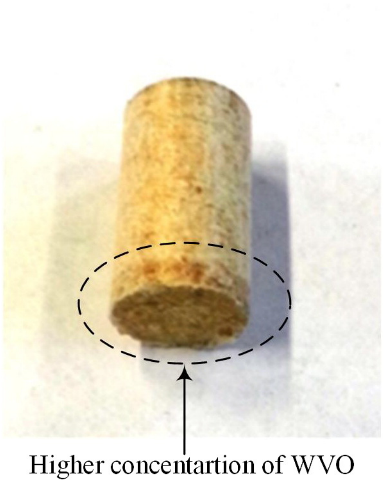

Fig. 9. Example of the 'oil gradient' in L_1 pellet produced at 150 MPa, 120 °C.

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